DETERMINATION OF THE VAPOR PRESSURE OF K32

FINAL REPORT

TESTING FACILITY: Wildlife International
(Now doing business as EAG Laboratories)

PROJECT NUMBER: 786C-102

U.S. Environmental Protection Agency. Product Properties Test Guidelines,
OPPTS 830.7950, Vapor Pressure
and
Organisation for Economic Cooperation and Development, Guideline for the Testing of Chemicals
OECD Guideline for Testing of Chemicals
OECD 104, Vapour Pressure

AUTHORS:
William R. Schutt, B.S.
Raymond L. Van Hoven, Ph.D.
Eric S. Bodle, Ph.D.

STUDY INITIATION DATE: September 23, 2016
STUDY COMPLETION DATE: December 2, 2016

SUBMITTED TO:

KOCH AGRONOMIC SERVICES, LLC
2883 Miller Road
Decatur, GA 30035
United States

WILDLIFE INTERNATIONAL
Is now doing business as EAG Laboratories
8598 Commerce Drive
Easton, Maryland 21601 USA

EAG LABORATORIES

GOOD LABORATORY PRACTICE COMPLIANCE STATEMENT

SPONSOR: KOCH AGRONOMIC SERVICES, LLC

TITLE: Determination of the Vapor Pressure of K32

EAG LABORATORIES PROJECT NUMBER: 786C-102

STUDY COMPLETION: December 2, 2016

This study was conducted in compliance with Good Laboratory Practice Standards as published by the U.S. Environmental Protection Agency in 40 CFR Part 792, August 17, 1989 and OECD Principles of Good Laboratory Practice (ENV/MC/CHEM (98) 17), with the following exception:

The stability of the test and reference substances at the test site was not determined in accordance with Good Laboratory Practice Standards.

STUDY DIRECTOR:

William D Schutt — 2 Dec 2016

William R. Schutt, B.S.
Senior Chemist
EAG Laboratories-Easton

Date

SPONSOR'S APPROVAL:

Eric C. Searcy — 12/2/2016

Sponsor's Representative
Eric Searcy

Date

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QUALITY ASSURANCE STATEMENT

This study was examined for compliance with Good Laboratory Practice Standards as published by the U.S. Environmental Protection Agency in 40 CFR Part 792, 17 August 1989 and OECD Principles of Good Laboratory Practice (ENV/MC/CHEM (98) 17). The dates of all inspections and audits and the dates that any findings were reported to the Study Director and Laboratory Management were as follows:

		DATE REPORTED TO:	
ACTIVITY:	DATE CONDUCTED:	STUDY DIRECTOR:	MANAGEMENT:
Protocol	September 28, 2016	September 28, 2016	September 28, 2016
Test Initiation	September 29, 2016	September 29, 2016	October 3, 2016
Data and Draft Report	October 10-13, 2016	October 13, 2016	October 24, 2016
Final Report	December 2, 2016	December 2, 2016	December 2, 2016

All inspections were study-based unless otherwise noted.

Mareeya Muneer — Dec. 2, 2016

Mareeya Muneer
Quality Assurance Supervisor
EAG Laboratories-Easton

Date

EAG LABORATORIES

REPORT APPROVAL

SPONSOR: KOCH AGRONOMIC SERVICES, LLC

TITLE: Determination of the Vapor Pressure of K32

EAG LABORATORIES PROJECT NUMBER: 786C-102

STUDY DIRECTOR:

William R. Schutt, B.S.
Senior Chemist
EAG Laboratories-Easton

2 Dec 2016
Date

MANAGEMENT:

Raymond L. Van Hoven, Ph.D.
Manager of Product Chemistry
EAG Laboratories-Easton

02 December 2016
Date

EAG LABORATORIES

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SUMMARY

STUDY TITLE:	Determination of the Vapor Pressure of K32
EAG LABORATORIES PROJECT NUMBER:	786C-102
SPONSOR:	KOCH AGRONOMIC SERVICES, LLC 2883 Miller Road Decatur, GA 30035 United States
TESTING FACILITY:	EAG Laboratories Easton, Maryland 21601
SPONSOR'S REPRESENTATIVE:	Eric Searcy
LOCATION OF STUDY, RAW DATA AND A COPY OF THE FINAL REPORT:	EAG Laboratories 8598 Commerce Drive Easton, Maryland 21601
TEST SUBSTANCE:	K32
TEST DATES:	Experimental OECD Start – September 28, 2016 Experimental EPA Start – September 28, 2016 Experimental Termination – October 4, 2016
SUMMARY:	The vapor pressure of K32 was determined at 20° ± 1°C using the static method. A single vapor pressure determination was performed on three separate aliquots of the test substance. The mean measured vapor pressure of K32 by the static method was 1798 ± 709 Pa (N = 3, CV = 39%) at 20° ± 1°C. The vapor pressure of a multicomponent material as determined by the static method would be reflective of the most volatile component, which based on the information provided would be water (17.5 Torr or 2333 Pa at 20°C) (3). The high CV is most likely attributed to the physical/chemical nature of the UVCB test material and the overall abundance of water in each test substance aliquot at the time of vapor pressure measurement.

INTRODUCTION

The vapor pressure of K32 was determined at 20° ± 1°C using the static method. EAG Laboratories conducted this study for Koch Agronomic Services, LLC at the EAG Laboratories analytical chemistry facility in Easton, Maryland. Vapor pressure analyses were conducted from September 28, 2016 to October 4, 2016. Raw data generated by EAG Laboratories and a copy of the final report is filed under Project Number 786C-102 in archives located on the EAG Laboratories-Easton site.

OBJECTIVE

The objective of this study was to experimentally determine the vapor pressure of K32 at 20° ± 1°C.

EXPERIMENTAL DESIGN

The static method is applicable to both liquids and solids with vapor pressures at or above the upper range of the spinning rotor gauge method (0.5 Pa). Measurements were performed using a Pirani capacitance diaphragm manometer. The vacuum system and manometer were isolated from the test substance, maintained at a temperature of 20° ± 1°C, until ultimate vacuum was achieved. The system and manometer were then isolated from the vacuum pumps and the test material was introduced into the evacuated system. The pressure increase was monitored by the capacitance manometer at approximately one-second intervals. A linear regression was obtained for pressure increase as a function of time. The y-intercept of this regression corresponds to the vapor pressure of the sample.

MATERIALS AND METHODS

The study was conducted according to the procedures outlined in the protocol, “Determination of the Vapor Pressure of K32” (Appendix 1). The test was performed based on procedures in the U.S. EPA Product Properties Test Guidelines, OPPTS 830.7950, *Vapor Pressure* (1) and OECD Guideline for Testing of Chemicals, 104, *Vapour Pressure* (2).

Test Substance

The test substance was received from Ricerca Biosciences, LLC on September 19, 2016 and was assigned testing facility identification number 13308 upon receipt. The test substance was described as a liquid and identified as: K32; Lot number: 5570-30-13. An expiration date was not provided. The test

substance was stored under refrigerated conditions in darkness. A Certificate of Analysis for the test material was provided by the Sponsor (Appendix 2) and provided the following information:

Description: Off white to pale yellowish gel
NBPT Content: 17.3 wt%
Water Content: 2.4 wt%

Reference Substance

The toluene reference substance was received from Sigma-Aldrich Chemical Company on September 22, 2016, and was assigned testing facility identification number 13315 upon receipt. The reference standard, a liquid, was identified as: Toluene anhydrous, 99.8%; lot number SHBG9996V; CAS number 108-88-3. The reference standard had a reported purity of 99.90%, and was stored in darkness under ambient conditions (Appendix 2). An expiration date of September 22, 2016 was assigned.

System Configuration

The high vacuum system consisted of a vacuum chamber configured with a mechanical vacuum pump (Edwards Model E2M2), a turbomolecular pump (Leybold TURBOVAC 50), a pirani capacitance diaphragm manometer (Agilent PCG-750), all-metal isolation valves, a sample chamber and miscellaneous vacuum fittings required to assemble the components. The pirani gauge controller was connected to a computer via the serial interface (RS-232 port). A diagram of the apparatus configuration and an image of the high vacuum system are presented in Figures 1 and 2, respectively.

The sample chamber consisted of a Pyrex tube (~2.5 cm o.d. X 12 cm) joined to a quick-fit type metal flange (NW-25). The temperature of the sample chamber was maintained at 20° ± 1°C during pressure measurements. A refrigerated recirculator, attached to a copper water jacket, regulated the temperature of the sample chamber. The minimum and maximum temperatures of the sample chamber during measurement were recorded with a NIST-traceable digital thermometer.

Test Procedure

Prior to analysis of the reference or test substance, the vacuum system, including the pirani diaphragm capacitance manometer, was isolated from the sample chamber and evacuated to achieve ultimate vacuum. Once the system had achieved ultimate vacuum, the vacuum system was isolated from the vacuum pumps and the reference or test substance was introduced into the evacuated system. The rate of pressure increase due to out-gassing and permeation in the presence of the reference or test material

was determined at least once per sample aliquot at 20° ± 1°C. Pressure increase was measured for at least fifteen minutes after introduction of the reference or test material.

A 10.0-mL aliquot of toluene was used as a vapor pressure reference to verify the accuracy of the system configuration. The vapor pressure of the reference substance by the static method was compared to the published values by the dynamic method (1). The vapor pressure was determined three times.

Three approximate 1g aliquots of the test substance, K32, were used for vapor pressure determination. A single vapor pressure determination was performed on each aliquot.

Calculations

Least-squares-fit linear regression analysis was performed on the vapor pressure measurements collected after the valves to the vacuum pump were closed to determine the out-gassing rate. A minimum of ten data points from the end of the vapor pressure curve with an average rate of change for the replicates of less than or equal to 0.10% was used for the linear regression of the test substance since it provided the best linearity. In the absence of sample, the slope of the regression would be equal to the out-gassing rate of the system, and the y-intercept would be considered the baseline or background pressure. With sample present, the sample vapor pressure was also taken as the y-intercept of the corresponding regression line.

RESULTS AND DISCUSSION

The mean measured vapor pressure of the reference substance, toluene, by the static method was 3025 ± 181 Pa (N = 3, CV = 6.0%) at 20° ± 1°C. The vapor pressure was consistent with published values (1). Individual and mean measured vapor pressures (y-intercept of linear regression function) and the corresponding out-gassing rates (slope of linear regression function) for the reference substance at 20° ± 1°C are presented in Table 1. Representative vapor pressure and linear regression plots for toluene are presented in Figures 3 and 4, respectively.

The mean measured vapor pressure of K32 by the static method was 1798 ± 709 Pa (N = 3, CV = 39%) at 20° ± 1°C. The test substance, K32, is classified as an UVCB (i.e. Unknown or Variable compositions, Complex reaction products and Biological materials) consisting of 17.3 wt% NBPT (i.e. N-(n-butyl)thiophosphoric triamide and 2.4 wt% water. According to the EPA OPPTS 830.7950

guideline, the static method for the determination of vapor pressure is applicable to multicomponent solids and liquids (1). The vapor pressure of a multicomponent material as determined by the static method would be reflective of the most volatile component, which based on the information provided by the Sponsor would be water (17.5 Torr or 2333 Pa at 20°C) (3). A single vapor pressure determination was performed on three separate aliquots of the test substance since during a non-GLP method development trial a significant drop in vapor pressure occurred at each vapor pressure determination of a single test substance aliquot due to the loss of the most volatile component (i.e. water). The high CV can most likely be attributed to the physical/chemical nature of the UVCB test material and the overall abundance of water in each test substance aliquot at the time of vapor pressure measurement. Individual and mean measured vapor pressures (y-intercept of linear regression function) and the corresponding out-gassing rates (slope of linear regression function) for the test substance at 20° ± 1°C are presented in Table 2. Representative vapor pressure and linear regression plots for K32 are presented in Figures 5 and 6, respectively.

CONCLUSIONS

The vapor pressure of K32 was determined at 20° ± 1°C using the static method. The mean measured vapor pressure of K32 by the static method was 1798 ± 709 Pa (N = 3, CV = 39%) at 20° ± 1°C. This vapor pressure is reflective of the water component in the test material (17.5 Torr or 2333 Pa at 20°C) (3).

REFERENCES

1. **U.S. Environmental Protection Agency.** 1996. Product Properties Test Guidelines, OPPTS 830.7950, *Vapor Pressure.*

2. **Organisation for Economic Cooperation and Development.** 2006. Guideline for Testing of Chemicals, 104: V*apour Pressure.*

3. *Water, B&J Brand*®; Product Information [Online]; **Honeywell.** 2015. https://labchemicals-honeywell.com/water-b-j-brandr-365 (accessed October 5, 2016).

Table 1

Vapor Pressure Measurements and Out-Gassing Rates for Toluene
by the Static Method at 20°C ± 1°C

Temperature (°C)	Measured Vapor Pressure (Pa)	Min/Max Temperature (°C)	Mean Measured Vapor Pressure (Pa)[1]	Standard Deviation (Pa)	Coefficient of Variation (CV)
	3231	20.0/20.2			
20	2954	19.6/20.1	3025	181	6.0%
	2890	19.7/20.0			

Literature value was 3.0×10^3 Pa and range was 2.9×10^3 Pa to 3.1×10^3 Pa using the dynamic method (1).

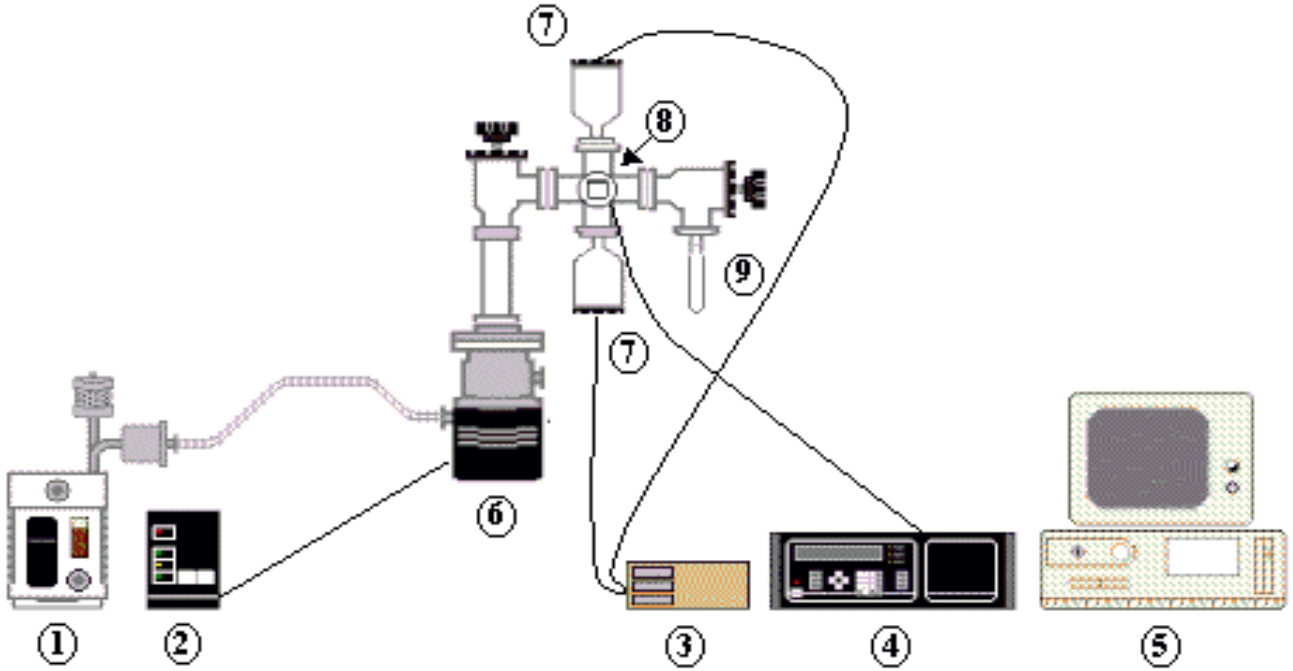
Temperature (°C)	Measured Out-gassing Rate (Pa/s)	Min/Max Temperature (°C)	Mean Measured Out-gassing Rate (Pa/s)	Standard Deviation (Pa/s)
	-2.30×10^{-2}	20.0/20.2		
20	2.08×10^{-1}	19.6/20.1	8.89×10^{-3}	1.9×10^{-1}
	-1.58×10^{-1}	19.7/20.0		

Table 2

Vapor Pressure Measurements and Out-Gassing Rates for K32
by the Static Method at 20°C ± 1°C

Temperature (°C)	Measured Vapor Pressure (Pa)	Min/Max Temperature (°C)	Mean Measured Vapor Pressure (Pa)	Standard Deviation (Pa)	Coefficient of Variation (CV)
	1965	20.2/20.4			
20	1020	20.0/20.3	1798	709	39%
	2409	19.9/20.1			

Temperature (°C)	Measured Out-gassing Rate (Pa/s)	Min/Max Temperature (°C)	Mean Measured Out-gassing Rate (Pa/s)	Standard Deviation (Pa/s)
	-2.05×10^{-2}	20.2/20.4		
20	5.10×10^{-1}	20.0/20.3	2.58×10^{-1}	2.7×10^{-1}
	2.84×10^{-1}	19.9/20.1		



1. Vacuum Pump
2. Turbopump Controller
3. Capacitance Manometer Controller
4. Spinning Rotor Gauge Controller (Not Used)
5. Computer Workstation
6. Turbomolecular Pump
7. Capacitance Manometer
8. Spinning Rotor Gauge (Not Used)
9. Sample Chamber

Figure 1. Diagram of high vacuum system configuration.

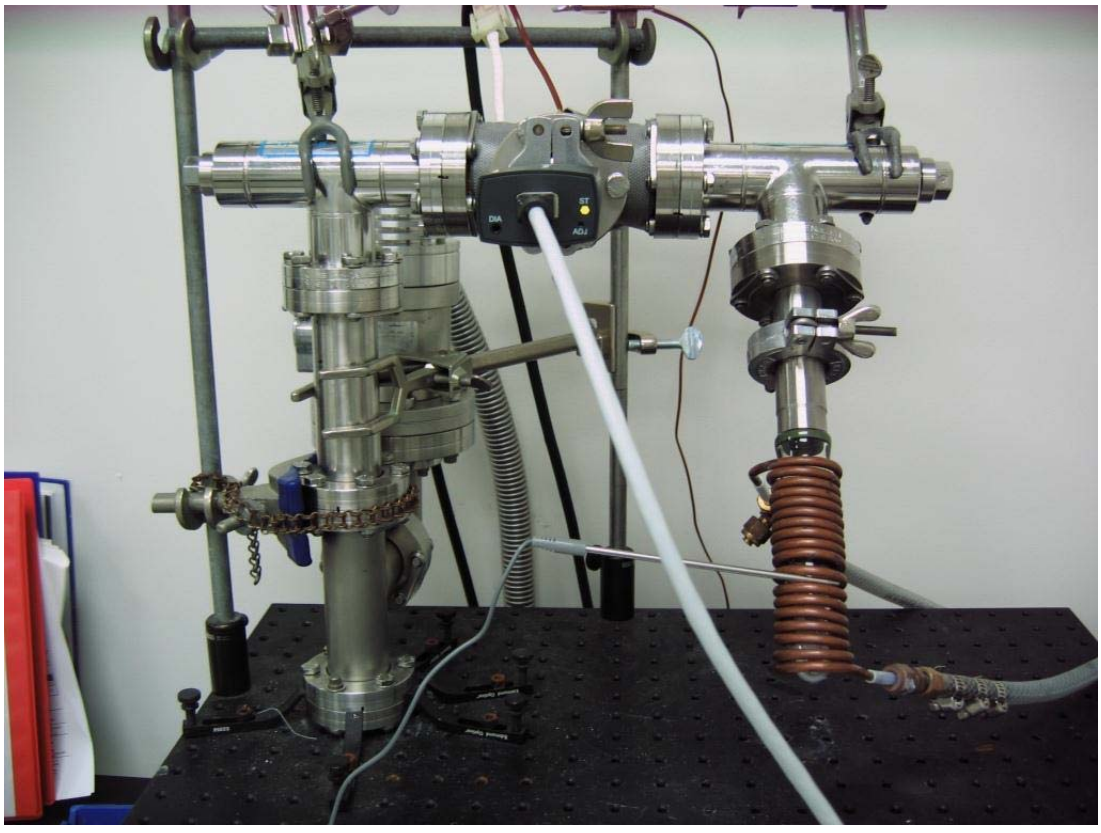


Figure 2. Image of the high vacuum system.

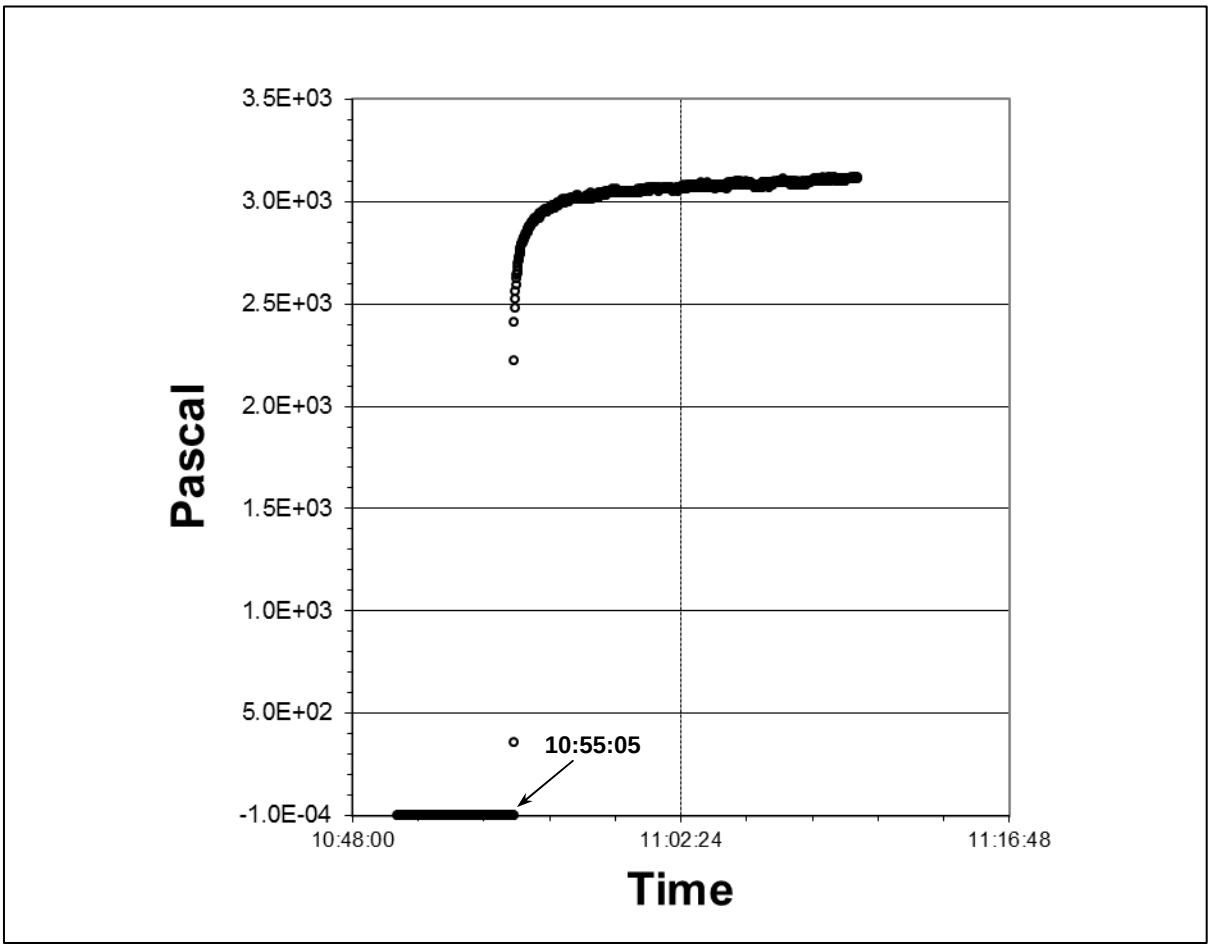


Figure 3. Representative vapor pressure plot for toluene at 20° ± 1°C. Arrow indicates time of valve closing (vacuum isolation) and introduction of sample into the evacuated system.

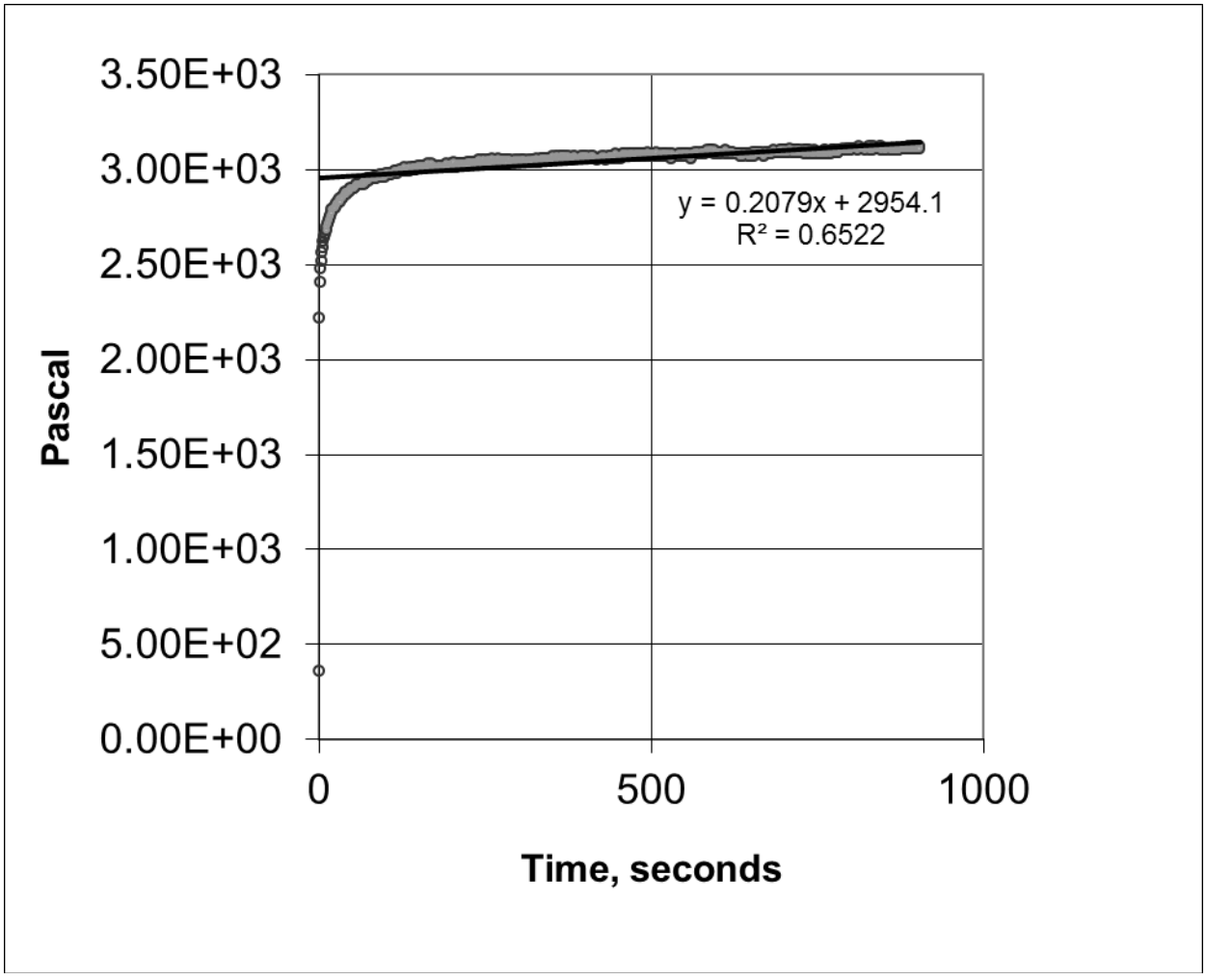


Figure 4. Representative linear regression plot for toluene at 20° ± 1°C.

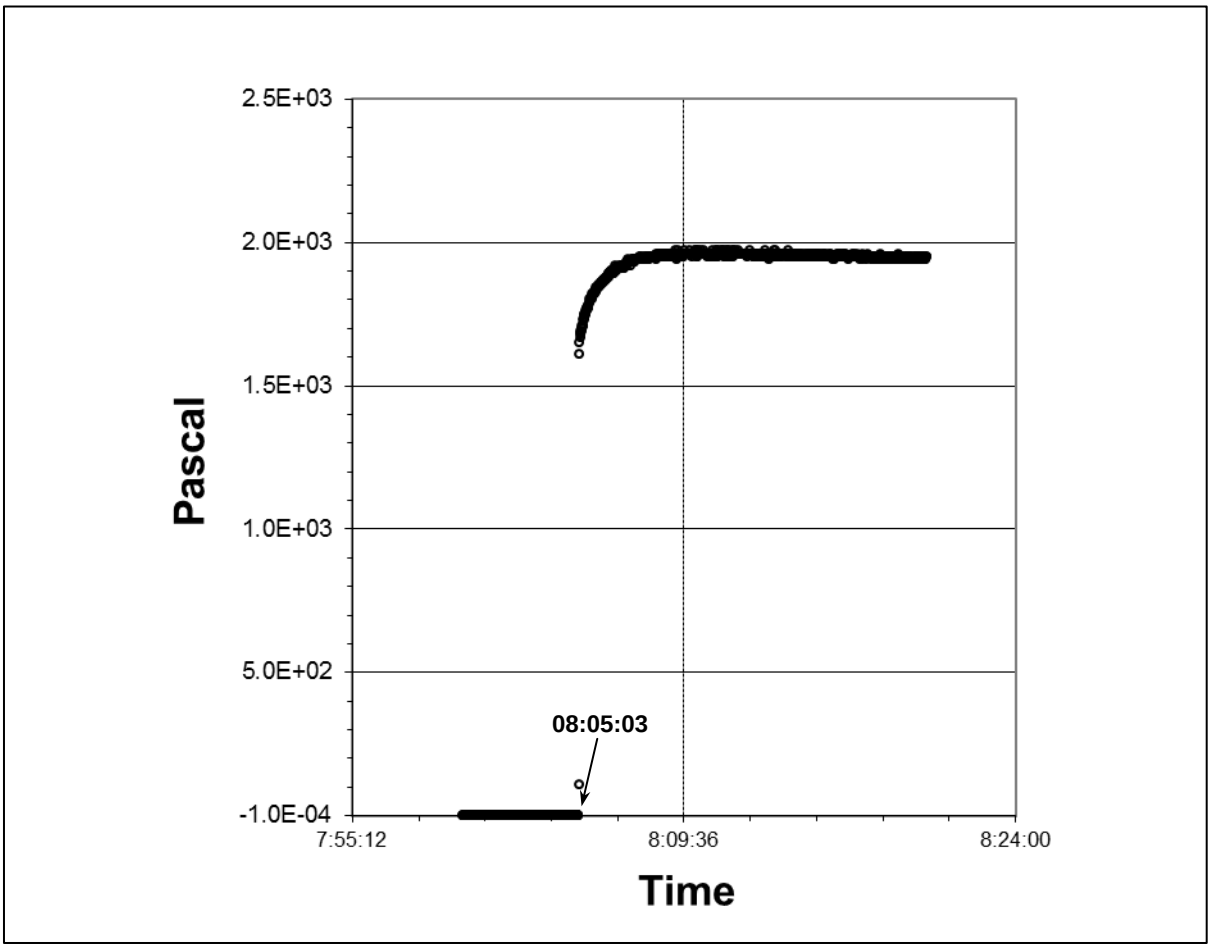


Figure 5. Representative vapor pressure plot for K32 at 20° ± 1°C. Arrow indicates time of valve closing (vacuum isolation) and introduction of sample into the evacuated system.

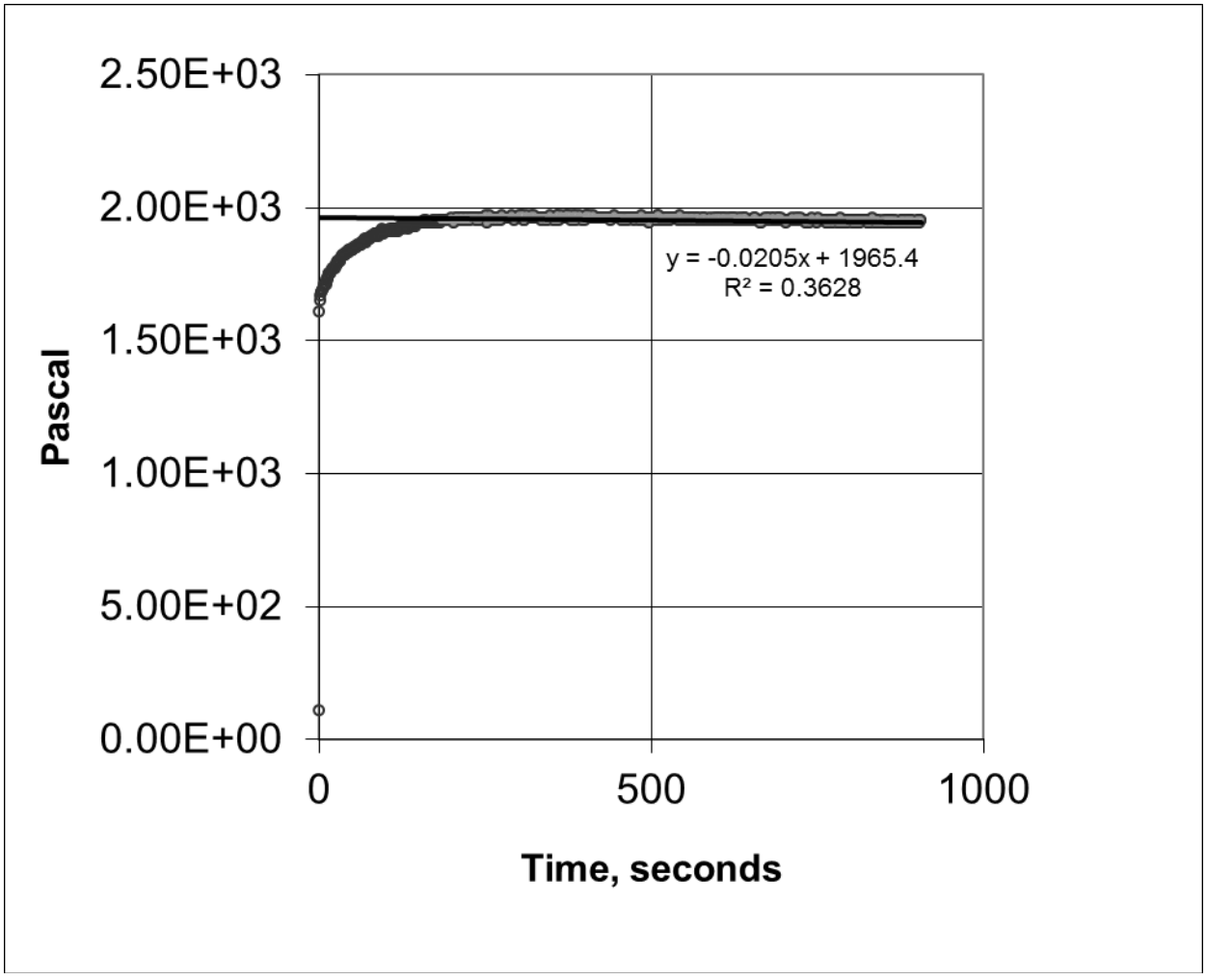


Figure 6. Representative linear regression plot for K32 at 20° ± 1°C.

Appendix 1

Study Protocol

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PROTOCOL

DETERMINATION OF THE VAPOR PRESSURE OF K32

U.S. EPA Product Properties Test Guidelines
OPPTS 830.7950, Vapor Pressure

and

OECD Guideline for Testing of Chemicals
OECD 104, Vapour Pressure

Submitted to

Koch Agronomic Services, LLC
2883 Miller Road
Decatur, GA 30035

EAG LABORATORIES

Formerly doing business as Wildlife International

8598 Commerce Drive
Easton, Maryland 21601 USA
1 (410) 822-8600

September 6, 2016

EAG LABORATORIES

DETERMINATION OF THE VAPOR PRESSURE OF K32

SPONSOR: Koch Agronomic Services, LLC
2883 Miller Road
Decatur, GA 30035

SPONSOR'S REPRESENTATIVE: Eric Searcy
Product Regulatory Manager
Koch Agronomic Services, LLC
2883 Miller Road
Decatur, GA 30035
Phone: 770-593-6813
Email: eric.searcy@kochind.com

TESTING FACILITY: EAG Laboratories
8598 Commerce Drive
Easton, Maryland 21601

STUDY DIRECTOR: William R. Schutt, Senior Chemist
EAG Laboratories-Easton

LABORATORY MANAGEMENT: Raymond L. Van Hoven, Ph.D.
Manager of Product Chemistry

FOR LABORATORY USE ONLY

Proposed Dates:
Experimental Start Date: 27 September 2016 Experimental Termination Date: 30 September 2016
Project No.: 786C-102
Test Substance No.: 13308 Reference Substance No(s). 13315

PROTOCOL APPROVAL

William Schutt — 23 September 2016
STUDY DIRECTOR — DATE

[signature] — 23 September 2016
LABORATORY MANAGEMENT — DATE

[signature] — 9/8/2016
SPONSOR'S REPRESENTATIVE — DATE

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INTRODUCTION

EAG Laboratories will determine the vapor pressure of the test substance at 20°C using either the spinning rotor gauge method or the static method. The study will be conducted at the EAG Laboratories analytical chemistry facility in Easton, Maryland. The study will be performed based on procedures in the OECD Guidelines for the Testing of Chemicals, 104, *Vapour Pressure* (1) and U.S. EPA Product Properties Test Guidelines, OPPTS 830.7950, *Vapor Pressure* (2).

OBJECTIVE

The objective of this study is to determine the vapor pressure of the test substance at 20 ± 1°C.

EXPERIMENTAL DESIGN

The spinning rotor gauge (SRG) method described in this protocol is applicable to both liquids and solids with vapor pressures in the range 1 x 10^{-4} Pascal (Pa) to 0.5 Pa. The SRG method is based on the measurement of the rotational frequency of a stainless steel ball, which is magnetically suspended within a vacuum chamber. In the presence of a sample in an isolated vacuum, the deceleration rate of ball rotation is proportional to the vapor pressure of the sample. Measurement is initiated with a determination of the offset value, system background pressure and out-gassing rate in the absence of sample. The background pressure and out-gassing rate are determined at least three times. The vacuum system and SRG are then exposed to the test substance, maintained at a temperature of 20° ± 1°C, until a steady state pressure is obtained. The sample and SRG are then isolated from the vacuum pumps and the pressure increase is monitored by the SRG at pre-set intervals (e.g. 30 seconds). A linear regression is obtained for pressure increase as a function of time. The y-intercept of this regression corresponds to the vapor pressure of the sample.

The static method described in this protocol is applicable to both liquids and solids with vapor pressures at or above the upper range of the spinning rotor gauge method (0.5 Pa). Measurements are performed using a Pirani capacitance diaphragm manometer. The vacuum system and manometer are isolated from the test substance, maintained at a temperature of 20° ± 1°C, until ultimate vacuum is achieved. The system and manometer are then isolated from the vacuum pumps and the test material is introduced into the evacuated system. The pressure increase is monitored by the capacitance manometer at approximately one-second intervals. A linear regression is obtained for pressure increase as a function of time. The y-intercept of this regression corresponds to the vapor pressure of the sample.

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MATERIALS AND METHODS

Test Substance

Information on the characterization of test, control or reference substances is required by Good Laboratory Practice (GLP) Standards and Principles. The Sponsor is responsible for providing EAG Laboratories verification that the test substance has been characterized prior to its use in the study. If verification of test substance characterization is not provided to EAG Laboratories, it will be noted in the compliance statement of the final report.

The Sponsor is responsible for all information related to the test substance and agrees to accept any unused test substance and/or test substance containers remaining at the end of the study.

Apparatus

The high vacuum system consists of a vacuum chamber configured with a mechanical pump (e.g. Edwards Model E2M2 or equivalent), a turbomolecular pump (Leybold TURBOVAC 50), isolation valves, a sample chamber and miscellaneous vacuum fittings. According to the expected vapor pressure of the test substance, the system will also include either a spinning rotor gauge (Leybold VISCOVAC VM212) or a pirani capacitance diaphragm manometer (Inficon PCG400). The appropriate gauge controller is connected to a computer via the serial interface (RS-232 port). The system configuration and components may be adapted as necessary to achieve high vacuum and sufficiently stable pressure readings to conduct the study. The system configuration utilized in the study will be described in the final report.

The sample chamber consists of a Pyrex tube (~2.5 cm o.d. X 12 cm) joined to a quick-fit type metal flange (NW-25). The temperature of the sample chamber is maintained at 20 ± 1°C during pressure measurements. A refrigerated recirculator, attached to a copper water jacket, regulates the temperature of the sample chamber. The minimum and maximum temperature of the sample chamber during measurement will be recorded using a NIST-traceable digital thermometer.

Test Procedure – Spinning Rotor Gauge Method

Prior to analysis of the test substance, the vacuum system, including the sample chamber, will be evacuated to achieve ultimate vacuum with no reference or test substance present in the sample chamber. The system may be heated, if necessary, to achieve adequate vacuum.

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An offset value (background deceleration rate) for the spinning rotor gauge will be determined at the system's ultimate pressure at the operating temperature used for the study. The sample chamber will be controlled at 20° ± 1°C during vapor pressure analysis. The rest of the apparatus may be at or above the target temperature.

The background pressure and the rate of pressure increase due to out-gassing and permeation will be determined at least three times at 20° ± 1°C. The steady state pressure of the vacuum system and the rate of pressure increase due to out-gassing and permeation in the presence of the test material will be determined at least three times. Background pressure and pressure changes will each be measured for at least five minutes prior to and after cycling the isolation valve to the pump.

A vapor pressure reference compound (e.g. benzoic acid, 0.04 to 0.07 Pa using the gas saturation and vapor pressure balance methods) will be used to confirm the system configuration and the measurement procedures. The measured vapor pressure of the reference material is not used for calculating the vapor pressure of the test substance, but may be used for comparison with other measurement methods.

Test Procedure – Static Method

Prior to analysis of the reference or test substance, the vacuum system, including the capacitance manometer, will be isolated from the sample chamber and evacuated to achieve ultimate vacuum. The system may be heated, if necessary, to achieve adequate vacuum.

Once the system has achieved ultimate vacuum, the vacuum system will be isolated from the vacuum pumps and the reference or test substance will be introduced into the evacuated system. The rate of pressure increase due to out-gassing and permeation in the presence of the reference or test material will be determined at least three times at 20 ± 1°C. Pressure increase will be measured for at least fifteen minutes after introduction of the reference or test substance.

A vapor pressure reference compound (e.g. toluene, 2.9 x 10^3 to 3.1 x 10^3 Pa using the dynamic method) will be used to confirm the system configuration and the measurement procedures. The measured vapor pressure of the reference material is not used for calculating the vapor pressure of the test substance, but may be used for comparison with other measurement methods.

Calculations

A linear regression (least squares fit) will be performed on the vapor pressure measurements collected after the valves to the vacuum pumps are closed to determine the out-gassing rate. The linear regression will consist of a minimum of ten data points from the end of the vapor pressure curve with an average rate of change for the replicates that would provide the best linearity. The out-gassing rate of the system will be equal to the slope of the regression equation in the absence of sample. The uncorrected vapor pressure of the sample will be equal to the intercept of the regression equation, i.e. the vapor pressure at the moment the valves are closed. The means and standard deviations of the vapor pressures and out-gassing rates will be calculated.

For vapor pressure determinations using the spinning rotor gauge method, it may be necessary to manually calculate the vapor pressure and/or offset for test substances with molecular weights greater than 999. The necessary equations and example calculations are presented in Appendix I.

Sample Handling and Safety

The Sponsor will identify any special handling or safety precautions to be used with the above referenced test substance. All normal precautions with respect to handling and storage will be taken.

Sample and Test Substance Retention

Upon completion of testing, portions of the test substance used as part of this study will be disposed of in accordance with federal, state and local regulations. Test substance containers and any unused portion of the test substance remaining at the end of the study will be retained by EAG Laboratories for use in other studies and then will be returned to the Sponsor.

RECORDS TO BE MAINTAINED

Records to be maintained for data generated by EAG Laboratories will include, but not be limited to:

1. A copy of the signed protocol.

2. Identification and characterization of the test substance, if provided by the Sponsor.
3. Dates of initiation and completion of the study.
4. Dates of experimental start and termination.
5. Storage conditions of the test substance.
6. Test substance use log.
7. Vapor pressure readings.
8. Statistical calculations.
9. Test conditions.
10. A copy of the final report.

FINAL REPORT

EAG Laboratories will prepare a final report of the results of the study. A draft final report will be provided to the Sponsor's Representative for review prior to issuance of a final report. The final report will include, but not be limited to the following, when applicable:

1. Name and address of the facility performing the study.
2. Dates upon which the study was initiated and completed.
3. A statement of compliance signed by the Study Director addressing any exceptions to Good Laboratory Practice Standards.
4. Purpose and procedure, as stated in the approved protocol, including all amendments and deviations to the protocol.
5. The test substance identification, including name, chemical abstract number or code number, purity, composition, empirical formula, molecular formula, manufacturer's lot/batch number, dissociation in water, method of analysis, or other information, if provided by the Sponsor.
6. Description of the test method or reference to the method used along with any modifications made. Individual and mean values obtained for out-gassing rates and vapor pressure.
7. Description of any problems experienced and how they were resolved.
8. A statement prepared by the Quality Assurance Unit listing the dates that study inspections and audits were made and the dates of any findings reported to the Study Director and Management.

If it is necessary to make corrections or additions to a final report after it has been accepted, such changes will be made in the form of an amendment issued by the Study Director. The amendment will clearly

identify the part of the final report that is being amended and the reasons for the amendment, and will be signed by the Study Director.

CHANGING OF PROTOCOL

Planned changes to the protocol will be in the form of written amendments signed by the Study Director and approved by the Sponsor's Representative. Amendments will be considered as part of the protocol and will be attached to the final protocol. Any other changes will be in the form of written deviations filed with the raw data. All changes to the protocol will be indicated in the final report.

GOOD LABORATORY PRACTICES

This study will be conducted in accordance with Good Laboratory Practice Standards for EPA (40 CFR Parts 160 and/or 792); and OECD Principles of Good Laboratory Practice (ENV/MC/CHEM (98) 17). Each study conducted by EAG Laboratories is routinely examined by the EAG Laboratories Quality Assurance Unit for compliance with Good Laboratory Practices, Standard Operating Procedures and the specified protocol. A statement of compliance with Good Laboratory Practices will be prepared for all portions of the study conducted by EAG Laboratories. The Sponsor will be responsible for compliance with Good Laboratory Practices for procedures performed by other laboratories. Raw data for all work performed at EAG Laboratories-Easton and a copy of the final report will be filed by project number in archives located on the EAG Laboratories site in Easton, Maryland or at an alternative location to be specified in the final report.

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REFERENCES

1 **Organisation for Economic Cooperation and Development.** 2006. Guidelines for the Testing of Chemicals, 104: *Vapour Pressure.*

2 **U.S. EPA Product Properties Test Guidelines.** 1996. OPPTS 830.7950. *Vapor Pressure.*

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Appendix I

Calculations

Calculation of Offset According to Molecular Weight

The offset is recorded as a deceleration rate of the freely rotating ball. When vapor pressure measurements are performed, this deceleration rate is converted to an offset vapor pressure in the corresponding units (Pa) according to the molecular weight of the material. This offset is subtracted from each measurement and the net difference is recorded as the vapor pressure of the test material. The SRG calculates this internally, but the offset can be calculated using the following equation:

$$\text{OFS (Pa)} = \frac{\text{BDIAM x BDENS}}{10 \times \sigma} \times \sqrt{\frac{2 \times \pi \times R \times \text{TEMP}}{\text{MOLWT}}} \times \frac{\text{DCR}}{1000}$$

Where:

BDIAM = Ball diameter (4.5 mm)
BDENS = Ball density (7,720 kg/m^3)
σ = Coefficient of friction (1.00)
R = Universal gas constant (8314 J/kmol/K)
TEMP = Test temperature (e.g. 293K)
MOLWT = Molecular weight
DCR = Deceleration rate (e.g. 7.1567 x 10^{-8} decelerations/second)
1000 = Conversion factor

Using the molecular weight of air (28.96), the offset for the background measurements would be calculated as follows:

$$\text{OFS (Pa)} = \frac{4.5 \times 7720}{10 \times 1.00} \times \sqrt{\frac{2 \times \pi \times 8314 \times 293}{28.96}} \times \frac{7.1567 \times 10^{-8}}{1000}$$

$$\text{OFS (Pa)} = 1.8075 \times 10^{-4}$$

Calculation of Vapor Pressure According to Molecular Weight

The SRG uses the reduction in speed of a freely rotating ball to determine the vapor pressure of a given material. The vapor pressure can be determined using the following equation:

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$$VP = \frac{BDIAM \times BDENS}{10 \times \sigma} \times \sqrt{\frac{2 \times \pi \times R \times TEMP}{MOLWT}} \times \frac{1}{t} \times \ln\left(\frac{v_0}{v_{(t)}}\right)$$

Where:

BDIAM = Ball diameter (4.5 mm)
BDENS = Ball density (7,720 kg/m^3)
σ = Coefficient of friction (1.00)
R = Universal gas constant (8314 J/kmol/K)
TEMP = Test temperature (e.g. 293K)
MOLWT = Molecular weight
t = Time (30 sec)
v_0 = Initial velocity
$v_{(t)}$ = Velocity at time t

Given that the molecular weight is the only variable that changes, the equation can be simplified as follows:

$$VP_{calc} = \sqrt{\frac{(VP_{meas})^2 \times MW_I}{MW_F}}$$

Where:

MW_I = Molecular weight used for vapor pressure measurements (999)
MW_F = Molecular weight of the test substance

Using this equation, an example calculated vapor pressure is given below.

Measured Vapor Pressure = 5.35×10^{-4} Pa
Instrumental Molecular Weight = 999
Actual Molecular Weight = 1157

$$VP_{1157} = \sqrt{\frac{(5.35 \times 10^{-4})^2 \times 999}{1157}}$$

$$VP_{1157} = 4.97 \times 10^{-4} \text{ Pa}$$

PROJECT NO.: 786C-102
Page 1 of 1

AMENDMENT TO STUDY PROTOCOL

STUDY TITLE: Determination of the Vapor Pressure of K32

PROTOCOL NO.: 786/090616/104-VP/100P-213 **AMENDMENT NO.:** 1

SPONSOR: Koch Agronomic Services, LLC **PROJECT NO.:** 786C-102

EFFECTIVE DATE: September 28, 2016

AMENDMENT:

Change: Page 4, Apparatus

From: Inficon PCG400
To: Agilent PCG-750

REASON: The Inficon gauge was not working properly and was replaced with the gauge from Agilent.

IMPACT: None. The Agilent gauge is an equivalent gauge.

William Schutt
STUDY DIRECTOR

13 October 2016
DATE

LABORATORY MANAGEMENT
Raymond L. Van Hoven, Ph.D.

13 October 2016
DATE

QAU Review
MM/ 10/13/2016
Initials/Date

Appendix 2

Certificates of Analysis

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Test Substance

ricerca
Ricerca means Research

CERTIFICATE OF ANALYSIS

(Page 1 of 1)

K32, Lot No. 55700-30-13

Material Name:	K32	Production Date:	July 20, 2016
Quantity Produced:	13.1 kg	Prepared By:	Ricerca Biosciences LLC

Test or Property	Test Result
Appearance	Off-white to pale yellowish gel
ID by ^{1}H NMR	Consistent with reference sample
NBPT Content (HPLC Assay, Avg of 3 preps)	17.3 wt%
Residual Ethyl Acetate (Estimated by ^{1}H NMR Analysis)	None detected
Water Content (Karl Fischer Analysis, Avg of 3 preps)	2.4 wt%

Data for this Certificate of Analysis are retained under Project Number 034689.

Recommended Storage Condition:	Refrigerated (2 - 8 °C)

7/20/16
Bill Karnofel
Associate Scientist II,
Process Chemistry
Date

7-20-16
Arthur Cooper
Director,
Process Chemistry
Date

Ricerca Biosciences, LLC • 7528 Auburn Rd. • Concord, OH 44077
Tel. 440.357.3300 • Fax 440.357.3654

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Reference Substance

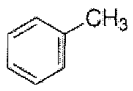
SIGMA-ALDRICH®

sigma-aldrich.com

3050 Spruce Street, Saint Louis, MO 63103, USA
Website: www.sigmaaldrich.com
Email USA: techserv@sial.com
Outside USA: eurtechserv@sial.com

Certificate of Analysis

Product Name:
Toluene - anhydrous, 99.8%



Product Number:	**244511**
Batch Number:	**SHBG9996V**
Brand:	SIAL
CAS Number:	108-88-3
MDL Number:	MFCD00008512
Formula:	C7H8
Formula Weight:	92.14 g/mol
Quality Release Date:	14 MAR 2016

Test	Specification	Result
Appearance (Color)	Colorless	Colorless
Appearance (Form)	Liquid	Liquid
Infrared Spectrum	Conforms to Structure	Conforms
Purity (GC)	≥ 99.75 %	99.90 %
Residue on Evaporation	≤ 0.0005 %	0.0001 %
Water (by Karl Fischer)	≤ 0.001 %	< 0.001 %
Note For 100 mL unit size, water content may exceed above specification; not to exceed 0.005%	----------	----------

Michael Grady, Manager
Quality Control
Sheboygan Falls, WI US

Sigma-Aldrich warrants, that at the time of the quality release or subsequent retest date this product conformed to the information contained in this publication. The current Specification sheet may be available at Sigma-Aldrich.com. For further inquiries, please contact Technical Service. Purchaser must determine the suitability of the product for its particular use. See reverse side of invoice or packing slip for additional terms and conditions of sale.

Appendix 3

Personnel Involved in the Study

The following key EAG Laboratories personnel were involved in the conduct or management of this study:

1. Eric S. Bodle, Ph.D., Director of Chemistry and Avian Toxicology
2. Raymond L. Van Hoven, Ph.D., Manager of Product Chemistry
3. William R. Schutt, B.S., Senior Chemist